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New polymer materials for the laser sintering process: polypropylene and others

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Abstract

Laser sintering of polymers gets more and more importance for small series production. However, there is only a little number of materials available for the process. In most cases parts are build up using polyamide 12 or polyamide 11. Reasons for that are high prices, a restricted availability, poor mechanical part properties or an insufficient understanding of the processing of other materials. These problems result from the complex processing conditions in laser sintering with high requirements on the material's characteristics. Within this area, at the chair for manufacturing technology fundamental knowledge was established.
Aim of the presented study was to qualify different polymers for the laser sintering process. Polyethylene, polypropylene, polyamide 6, polyoxymethylene as well as polybutylene terephthalate were analyzed. Within the study problems of qualifying new materials are discussed using some examples. Furthermore, the processing conditions as well as mechanical properties of a new polypropylene compound are shown considering also different laser sintering machines.

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Keywords: Laser sintering; mechanical properties; part's density; process understanding; plastics; polypropylene; polyamide 6; polyethylene; polybuthylen terephthalate; additive manufacturing

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Nomenclature

DSC	Differential scanning calorimetry
PA 6	Polyamide 6
PA 12	Polyamide 12
PBT	Polybutylene terephthalate
PE	Polyethylene
POM	Polyoxymethylene
PP	Polypropylene
EAB	Elongation at break

1. Introduction

Increasing competition, decreasing product life cycles, the wish for customized products and a shortage of resources cause the need for innovative manufacturing techniques for small series production, Abele and Reinhardt (2011). Going beyond the stage of Rapid Prototyping on to Rapid Manufacturing, Additive Manufacturing offers possibilities for small series production of customized products and an increased freedom of design due to the lack of tools, Hague et al. (2003). The laser sintering of plastic parts is, aside from beam melting of metal parts, one of only two AM-processes which have the capability to be used for Rapid Manufacturing in the near future, VDI 3405 (2014). Laser sintered parts are built up layer by layer. The machine produces the parts by repeating four stages for each layer: Firstly, the platform descends by the thickness of one layer. Secondly, powder is spread across the build platform by a leveling roller or coater. Thirdly, the layer is preheated to a temperature close to the material's melting point by a radiant heater. Then, a CO_2-laser beam melts the powder by tracing the actual cross section line after line using a scanner system. These steps are repeated until the parts are completed, Gebhardt (2007).

Laser sintering has reached a high technical level within the past two decades. However, there is only a little number of materials available for the process. In about 90 to 95 % of all cases parts are build up using polyamide 11 or polyamide 12 (PA 12), Goodridge et al. (2012) and Schmid (2015). Alternative materials are seldom used. Reasons for that are high prices, a restricted availability, poor mechanical part properties, a more difficult processing or an insufficient understanding of the processing. The problems with other materials result from the very complex processing conditions in laser sintering with high requirements on the material characteristics. Therefore, it is very complicated to develop new materials with good processing behavior, good mechanical properties as well as a sufficient ductility of the parts. Within this area, fundamental knowledge was established at the chair for manufacturing technology within the last years. Aim of the presented study is to qualify a polypropylene (PP) material for the laser sintering process. Additionally, problems of qualifying new materials are discussed using some other material examples.

2. State of the Art

Technical literature contains several papers with experiments on new materials. Rietzel analyzed in Rietzel (2011) polyoxymethylene (POM), polypropylene (PP), polybutylene terephthalate (PBT) and polyethylene (PE). Fiedler performed different experiments with PP in Fiedler et al. (2007) and Fiedler et al. (2008). Goodrigde and Khalil studied UHMWPE in Goodridge et al. (2010) and Khalil et al. (2016). Fraunhofer Umsicht and RPM evaluated the processing of PBT in Gerken (2009) and Bertling and Eloo (2009). Drummer developed in Drummer et al. (2012) and Wudy et al. (2015) new PA12/PP as well as POM/PP blends for the laser sintering process. Additionally, some studies consider the development of new powder production processes like the powder spraying using overcritical CO_2 in Bertling and Eloo (2009), the use of immiscible blends to produce PA 12 powder in Drummer et al. (2015), the melt emulgration of polypropylene powder in Fanselow et al. (2015). However, none of these studies led to new commercial materials.

Reasons for that are the very complex processing conditions in laser sintering. These result in high requirements on the powder materials as discussed in Rietzel (2011), Alscher (2000) and Schmid et al. (2014). Material should have a wide processing window between the beginning of melting processes when heated up and the start of the crystallization process when cooled down again. Crystallization speed should be as low as possible in order to avoid warpage. The melt of the materials should have a suitable rheology and surface tension in order to form a flat film after laser exposure. The used powders need to have good flowing properties and preferable round particle shape in order to allow good powder spreading during the process. In addition to that, powders should have high bulk density and a suitable powder size distribution for the laser sintering process. Furthermore, the material should have a high absorption of the CO_2 laser beam wavelength of 10.6 µm.

Rietzel studied in Rietzel (2011) the thermal and powder properties, the processing conditions as well as the mechanical properties of different materials like PE, POM, PBT or PP. The analyzed PE and PP material was produced by precipitation while the POM and PBT powder was made by cryogenic grinding. The named powders had a percentaged bulk density fewer than 40 % oftentimes even under 35 % while standard laser sintering PA 12 has values over 40 %. Also the powder flowability showed worse values for the PP, PE and POM materials compared to PA 12 while the flowability of PBT is only a little bit lower than for PA 12. Additionally, Rietzel analyzed the processing windows of the different materials between melting point and crystallization point (ΔT_{pm}-T_{pc}) by differential scanning calorimetry (DSC). PA 12 (32.8 K) has a window twice the size of POM (14.4 K) and nearly four times larger than PE (8.7 K). In contrast to that, PP has even a bigger processing window of 39.6 °C. All materials considered were usable to produce parts in the laser sintering process. Only PBT was not tested within the study. However, all tested materials show low values for elongation at break (EAB). For none material more than 6 % EAB was achieved, Rietzel (2011).

Fiedler published some papers on the development of a polypropylene laser sintering material: Fiedler et al. (2007), Fiedler et al. (2008), Radusch et al. (2011) and Fiedler et al. (2010). He studied different PP homopolymers and copolymers having varying material properties. While doing so, he preferred materials which were available as powder. Basing on the different studied materials he developed PP compounds being a homopolymer which was modified with a copolymer. He used contents of the copolymer between 8 and 50 %. With these materials he made laser sintering tests using a DTM Sinterstation 2500. Therefore, he used a grinded powder made from the developed compounds. Like in the study from Rietzel also the results of Fiedler show a brittle part behavior with an elongation at break under 5 %. Additionally, also tensile strength was found to be significantly under values of injection molded part from the same material.

Reinhardt considered in Reinhardt et al. (2013) a commercial glass-filled PP material: Microfol Sinterplast PP. This material was available on the market for some years. However, it was withdrawn from the market due to unknown reasons. Reinhardt studied the effects of different processing parameters of an EOS Formiga P100 on the different part properties considering also the part orientation. He optimized the parameter settings in order to achieve optimal part properties. However, even for optimized parameters parts show a low elongation at break of under 7 % in x-direction. Kleijnen studied in Kleijnen et al. (2015) another commercial laser sintering material from Diamond Plastics PP CP 22. However, mechanical properties are very low even when considering different energy density levels. EAB in x-direction is under 1 % while ultimate tensile strength is under 12 N/mm². Schmid analyses in Schmid et al. (2011) another PP material named iCoPP which shows superior part properties and very high elongation at break. The values are going up to over 400 % for reused material while virgin material shows values between 75 to 150 % EAB. Young's modulus is about 900 N/mm² while tensile strength is circa 21 N/mm². However, material has a high price and a limited availability. Therefore, the use for laser sintered is restricted. The same low-isotaciy material from Trial Corporation was studied in Zhu et al. (2015) and Zhu et al. (2016). They analyzed different material properties and found that the powder particles are nearly ideal spherical. Additionally, they studied part density and mechanical properties as a function of energy input. A high energy input over 0.03 J/mm² is necessary to achieve dense parts. A further study from Lexow and Drummer characterize two different LyndellBassell polypropylene materials which were converted to powder by grinding, Lexow and Drummer (2016). They analyze the effect of antistatic agent as well as flow agent on the material properties and the processing of the produced powder materials. Results show good flow times. However, bulk density is under 39 %. The material modified with flow and antistatic agent has good processing conditions while mechanical properties are still low. Tensile strength is only 10 N/mm² and elongation at break is even under 1.3 %.

State of the art gives much information on the optimal characteristics of materials for laser sintering. There are also several studies on the development of new materials. However, it seems very difficult to transfer the given requirements to the development of powders. Therefore, parts show low mechanical properties or materials are too expensive. Within the work presented here a further study on material properties of different new materials is performed. Additionally, the part properties of a new developed polypropylene laser sintering material are studied. Correlations between processing parameters and part properties should be established considering also two different laser sintering machines.

3. Experimental Setup

Based on the aforementioned state of the art, the analyses were planned. The materials analyzed are a PE compound named Rolaserit PEGR and a PP compound named Rolaserit PP developed for laser sintering in cooperation between the chair for manufacturing technology and the ROWAK AG. ROWAK is an expert in powder conversion producing powders with good bulk properties, flowability and an adjusted powder size distribution. The further considered materials are a DuPont POM Homopolymer, a Lanxess PA 6 and a Lanxess PBT. These unmodified standard granules were converted to fine powders by grinding. Some material details are summarized in Table 1.

Table 1. Material properties (data sheet values).

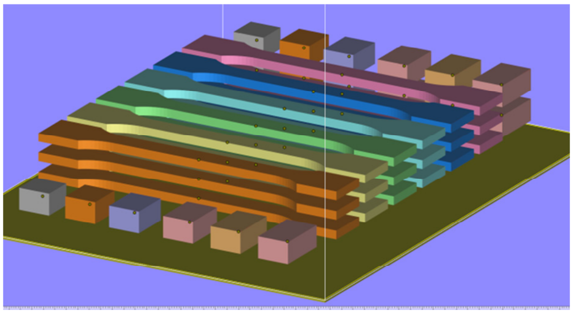
Material	Material properties	
	Melting Point [°C]	Density [g/cm³]
ROWAK Rolaserit PEGR	> 125	> 0.92
ROWAK Rolaserit PP	> 125	> 0.85
DuPont POM	178	1.42
Lanxess PA 6	222	1.14
Lanxess PBT	225	1.30

For all materials processing window (ΔT_{pm}-T_{pc}) was determined using differential scanning calorimetry according to DIN EN ISO 11357-1 and -3 using a Mettler Toledo DSC1/700. Heating rate was chosen to 20 K/min and cooling rate to 2 K/min in order to adjust the measurement on processing conditions in laser sintering with high heating rates due to the laser input and a slow cooldown process of the parts in the powder bed. In a second step the percentaged bulk density (bulk density/ material density) was determined according to DIN EN ISO 60. Furthermore, the Hausner-ratio (tap density/ bulk density) was determined measuring additionally the tap density.

Table 2. Processing parameters.

Machine	Parameters			
	Laser power [W]	Hatch distance [mm]	Scan speed [mm/s]	Volume energy density [J/mm³]
EOS Formiga P100				
Parameter 1	18	0.25	4000	0.18
Parameter 2	21.5	0.2	5000	0.22
Parameter 3	25	0.2	5000	0.25
Parameter 4	21.5	0.15	5000	0.29
Parameter 5	18	0.15	4000	0.3
Parameter 6	25	0.15	4000	0.42
DTM Sinterstation 2500				
Parameter 1	21	0.2	5000	0.21
Parameter 2	25	0.2	5000	0.25
Parameter 3	29	0.2	5000	0.29
Parameter 4	33	0.2	5000	0.33
Parameter 5	37	0.2	5000	0.37
Parameter 6	41	0.2	5000	0.41

a



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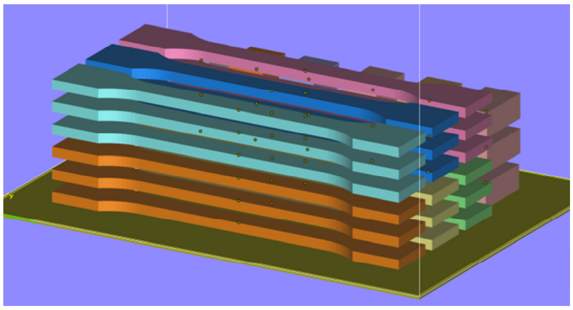

Fig. 1. Layout of the build process: (a) EOS Formiga P100; (b) DTM Sinterstation 2500.

After characterization of material and powder properties the PP material was tested in the laser sintering process using a layer thickness of 0.1 mm. The PP compound was processed on an EOS Formiga P100 as well as on a DTM Sinterstation 2500 with a build space reduction. Variation of parameters was done by changing volume energy density. Energy density is often used to describe correlations in laser sintering. This value for the energy input was introduced by Nelson (1993) and extended by Starr et al. (2011) and Kaddar (2010) with the layer thickness to define the volume energy density. It is defined as the ratio of fill laser power divided by beam speed, scan spacing and the layer thickness. For the PP energy density was varied between 0.18 and 0.42 J/mm³. Detailed parameters are shown in Table 2. Variation of hatch distance and scan speed for the Formiga results from a bigger setup while only six parameters were evaluated within this study. Processing temperatures were optimized before performing the tests and set to 127 °C (Formiga) and 125 °C (Sinterstation).

Tensile specimens according to DIN EN ISO 3167 and density cubes were produced for testing. Tensile bars were produced in x-direction. The tensile tests in compliance with DIN EN ISO 527-1 and DIN EN ISO 10350-1 were performed using a Zwick Z020 M (MutiXtense) and a testing speed of 5 mm/min. Part's density was measured for cubes of 20 x 20 x 10 mm size using the Archimedes method according to DIN 1183. Three parts were produced for tensile testing as well as density measurement.

4. Results and Discussion

4.1. Material Properties

The material properties were measured according to the methods described in chapter 3. The properties of six different materials are studied while two are commercial materials and the other four are experimental materials. The results are summarized in Table 3 using values for PA 12 standard laser sintering material from EOS (PA 2200) as a reference. The processing window (temperature difference between melting peak and crystallization peak) is for all analyzed materials smaller than for the PA 2200 reference. The processing window of PA 6 is with 32.9 K the biggest found for the alternative materials. The material shows during processing no problems like warpage. However, it was not possible to produce dense parts using that material yet. PBT has a processing window of 26.9 K. During processing different problems like warpage and cracks occur. Temperature windows of PA 12, POM and PE are bigger than the results found in Rietzel (2011). A reason for that might be the use of other material types and due to changed measuring conditions. However tendencies are similar for POM and PE. For POM the temperature range is with 20.0 K 40 % smaller than that for PA 12 while for PE the window is only 11.2 K. Therefore, usable temperature ranges for both materials will be smaller than for PA 12. Additionally, stronger warpage may occur. This is especially a problem when processing POM while PE show little warpage. This is surprising due to the point that PE has the smallest processing window of all studied materials. In contrast to PE and POM, the Rolaserit PP shows a different behavior. Nominal processing window is significantly smaller than for the material studied by Rietzel and also smaller than for PA 12. However, processing of the materials shows no problems, a large variety of possible processing temperatures as well as little warpage. Reason for that might be that a PP-compound was used within this study.

Table 3 shows additionally different powder properties of the studied materials. Rolaserit PEGR and Rolaserit PP have similar Hausner-ratios of 1.18 respectively 1.17 compared to the standard PA 12 with 1.15. Also the studied PA 6 has a low Hausner-ratio of 1.21. All three show a good flowability with a Hausner-ratio below 1.25. Additionally, during processing of these materials a smooth powder bed surface can be achieved. The values for percentaged bulk density of PE lie with 41.3 % only a little bit under the reference value of PA 12 (44.1 %) but much higher compared to the experiments in Rietzel (2011). Therefore, flowability and bulk density of the PE are suitable for laser sintering. The values of the Rolaserit PP (46.4 %) and the PA 6 (46.9 %) are even higher than the reference value resulting in a high density of packed powder bed. In contrast to that powder properties of the analyzed POM and PBT are worse compared to the other four materials. Hausner-ratio has a value of 1.30 respectively 1.33 which results in a reduced powder flowability. Percentaged bulk density is 38.8 % respectively 34.2 % and therefore over 12 % respectively 22 % lower than for PA 12. The reduced flowability and low bulk density negatively influence the powder spreading like processing tests of POM and PBT shows. This results in a coarser powder bed surface and a low packing density of powder bed.

Table 3. Material properties.

Material	Measured properties		
	Processing window (ΔT_{pm}-T_{pc}) [K]	Percentaged bulk density [%]	Hausner-ratio
PA 2200	34.9	44.1	1.15
Rolaserit PEGR	11.2	41.3	1.18
POM	20.0	38.8	1.30
PA 6	32,9	46.9	1.21
PBT	26.9	34.2	1.33
Rolaserit PP	27.3	46.4	1.17

Some results on part properties and processing of PE and POM were published in Wegner and Witt (2015a), Wegner and Witt (2015b) and Wegner (2016). This study will focus on the processing of Rolaserit PP and give some first results on part properties and process correlations.

4.2. Process correlations Polypropylene

Within the study the influence of energy input on different part properties were analyzed. Therefore, correlations between energy density and part density as well as tensile properties are established while two different laser sintering systems were compared.

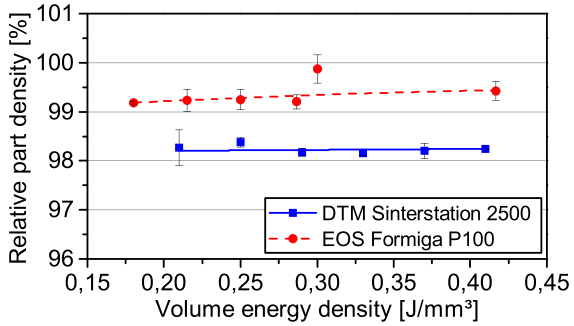

Fig. 2. Part density of Rolaserit PP as a function of energy density for two different laser sintering machines.

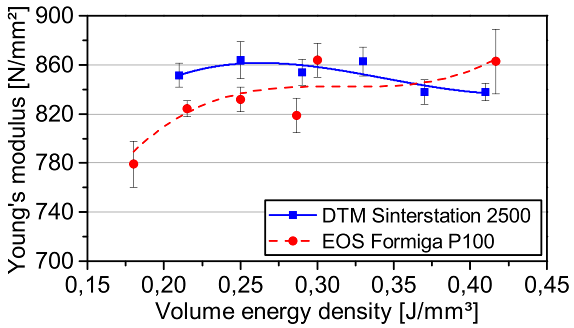

Fig. 3. Young's modulus of Rolaserit PP as a function of energy density for two different laser sintering machines.

Parts density of the Rolaserit PP shows only little variation within the studied energy density range, Fig. 2. However, there is a little difference between the two machines. For the Formiga P100 relative part density lies over 99 % which are very good values for laser sintered parts. In comparison, values for the DTM Sinterstation 2500 are little bit lower in the range of 98 to 98.5 % which is still good in laser sintering. The differences between the density values of both machines might be caused by the massive roller used in the Sinterstation. This massive roller leads to a bigger temperature reduction during powder spreading. This may result in a higher crystallinity of parts produced on the Sinterstation and therefore in a higher density. Same effect was found for PA 12 before in Wegner (2015) while comparing also both machine types. In conclusion, high part density comparable to other commercial laser sintering materials can be achieved for the ROWAK Rolaserit PP.

Young's modulus shows different correlations to the energy density, Fig. 3. For the DTM Sinteration 2500 young's modulus shows maximum values of ca. 860 N/mm² for energy densities between 0.21 and 0.33 J/mm³. Above that range, a little drop of values by 2.3 % is found. Young's modulus is then only 840 N/mm². Results for the EOS Formiga P100 show another behavior. Young's modulus increases for rising energy input. Lowest values of 798 N/mm² are achieved for the lowest energy input. Maximum values of circa 860 N/mm² are found for the two highest energy densities of 0.3 and 0.42 J/mm³. These are comparable to the values measured for the Sinterstation. However, higher energy input is necessary to achieve same level of young's modulus. Additionally, values for the Formiga show for some experiments significantly higher standard deviation as found for the DTM. Furthermore, values show bigger deviation to the fitted polynomial trend line. Fig. 4 shows the correlation between the tensile strength and the volume energy density. For the Sinterstation energy input shows only little effect on tensile strength. Values vary between 18 and 18.5 N/mm². The correlation for the Formiga shows a different trend. Tensile strength rises significantly for increasing energy input until a limit of 0.25 J/mm³. Over that limit values of ca. 17.5 to 18 N/mm² for the x-direction can be achieved. Results show again, that for the Formiga a higher energy input is necessary to get maximum values. However, these are 3 to 5 % lower than for the DTM machine. Reason for that might be again the different cooldown of the molten layer during powder spreading. Robust values for tensile strength with little effect of energy input can be established for both machines. Like for young's modulus, measured tensile strength for the Formiga again shows higher standard deviation.

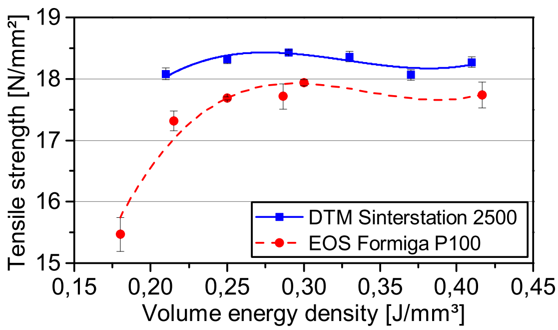

Fig. 4. Tensile strength of Rolaserit PP as a function of energy density for two different laser sintering machines.

Fig. 5 shows the correlation between the elongation at break (EAB) and volume energy density. Correlations are very different to the behavior found before. In contrast to the other mechanical properties, EAB shows a significant rise of values when energy density is increased. For the Sinterstation values increase from 11.7 % to over 15 %. Maximum values are achieved for energy densities over 0.37 J/mm² and therefore for the highest considered energy input. When results are compared to young's modulus, maximum values can only be reached for a reduction of young's modulus. The correlation for the Formiga shows also another behavior as found before. Values increase significantly from 8 % to 13 % when energy density is increased from 0.18 to 0.25 J/mm³. However, a drop of EAB by 1 to 2 % can be found for energy densities over 0.35 J/mm³. Optimal values between 11.5 and 13.2 % are achieved for energy densities between 0.25 and 0.35 J/mm³. When compared to the other studied properties all show optimal part properties for the Formiga within that range. However, also for EAB a significant difference between the two machines can be found. Maximum EAB values achieved for the DTM are about 15 % higher than the values of the Formiga. This is in contrast to the results for density and tensile strength where a higher crystallinity of the parts seems to cause the higher values for the Sinterstation. EAB show an opposite behavior. Parts with potential higher crystallinity have higher EAB. Reason for that behavior might be a difference in surface structure. This has to be evaluated in the future.

In conclusion, the measured maximum EAB values are comparable to standard PA 12-values which range from 15 to 23 %, Wegner and Witt 2015b. Therefore, the new developed polypropylene material has not the typical brittle behavior as found for many other material developments in laser sintering (compare chapter 3).

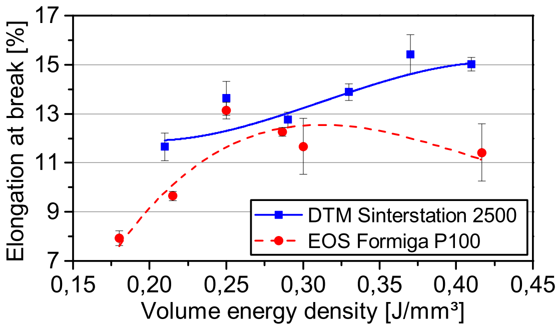

Fig. 5. Elongation at Break of Rolaserit PP as a function of energy density for two different laser sintering machines.

5. Conclusions

The powder flowability as well as the processing window of six different commercial and experimental laser sintering materials was studied. It was found that a larger processing window do not always guarantee for good processing in laser sintering. In contrast to that, good powder flowability is very important for a good powder spreading during the process. For one of these materials ROWAK Rolaserit PP, further analyses on the effect of processing conditions on part properties were done. Therefore, processing conditions on two very different laser sintering machines were compared. High part densities as well as good mechanical properties especially for elongation at break are achieved for the new polypropylene material.

Future work should improve the processing conditions of the studied experimental materials. Aim are the establishment of a good powder flowability, good processing conditions as well as good mechanical properties. Therefore, further analyses especially on PA 6 and PBT should be performed. For the polypropylene robust processing conditions were established yet. Future analyses should consider other part properties and especially aging effects during processing.

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